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Kinetics study on conventional and microwave pyrolysis of moso bamboo

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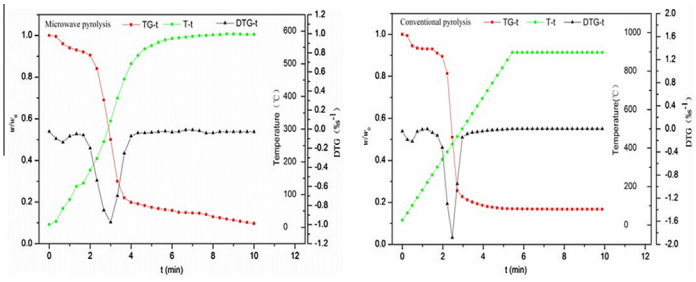
HIGHLIGHTS

- The heating rate had a significant effect on the moso bamboo pyrolysis process.
- Microwave heating led to a lower activation energy than conventional process.
- A kinetic compensation effect for moso bamboo pyrolysis was observed.

GRAPHICAL ABSTRACT



A comparison of moso bamboo pyrolysis processes between microwave and conventional conditions at a similar heating rate

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ABSTRACT

A comparative study on the pyrolysis kinetics of moso bamboo has been conducted in a conventional thermogravimetric analyzer and a microwave thermogravimetric analyzer respectively. The effect of heating rate on the pyrolysis process was also discussed. The results showed that both the maximum and average reaction rates increased with the heating rate increasing. The values of activation energy increased from 58.30 to 84.22 kJ/mol with the heating rate decreasing from 135 to 60 °C/min during conventional pyrolysis. The value of activation energy was 24.5 kJ/mol for microwave pyrolysis, much lower than that for conventional pyrolysis at a similar heating rate of 160 °C/min. The pyrolysis of moso bamboo exhibited a kinetic compensation effect. The low activation energy obtained under microwave irradiation suggests that microwaves heating would be a promising method for biomass pyrolysis.

1. Introduction

Pyrolysis is acknowledged as an attractive thermal–chemical method to realize the efficient conversion of biomass to high value-added products. Depending on the target product, the maximum production of oil, char or gas can be achieved by means of optimizing conditions such as temperature, heating rate, residence time, etc. (Dominguez et al., 2007). Currently, biomass pyrolysis process has been extensively studied in different prototype units, such as fluidized bed, fixed bed, rotating cone, transported bed, augur or screw (Bridgwater, 2012; Motasemi et al., 2014). However, all the above processes were conducted in conventional heating way, in which heat is transferred indirectly and slowly from the surface to the center of the material via conduction, convection and radiation. Therefore, the primary volatiles released from the material center must pass through the high temperature surface, which will inevitably cause undesired secondary reactions to occur (Miura et al., 2004).

Compared to conventional heating, microwave heating means the transfer of electromagnetic energy to thermal energy (Fernández et al., 2011). Due to the ability of microwaves to penetrate the material and deposit energy, heat can be generated throughout the volume of the material. Therefore, uniform and

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rapid heating of the material can be achieved during microwave pyrolysis. In consequence, microwave heating can result in a more uniform temperature distribution within sample than conventional heating. This is bound to result in different pyrolysis processes. For example, a uniform temperature distribution can prevent undesired secondary reactions, which can lead to a better control of the pyrolysis process and the attainment of desired end products (Zhao et al., 2012). Microwaves have been applied to pyrolyze biomass materials during the past decade, and the results showed that microwave pyrolysis could generate higher quality products compared with conventional heating (Beneroso et al., 2013; Dominguez et al., 2007, 2005; Miura et al., 2004; Yagmur et al., 2008).

Bamboo is a native plant to many Asian countries. It is one of the most rapid growing plants with a maturity cycle of 3–4 years (Hiziroglu et al., 2005). The bamboo resources are very abundant in China with the total plantation area being about 5 million hectares, among which moso bamboo has the largest plantation area and annual yield compared with all other kinds of bamboo (Jiang et al., 2012). Its great potentiality as a bio-energy resource has been recognized in the last decade. Most studies were carried out focused on the production of bamboo charcoal with high adsorption capacity. Only a small number of researches were conducted to assess the liquid and/or gaseous products obtained from bamboo pyrolysis (Jung et al., 2008; Qi et al., 2006; Xiao et al., 2007).

Kinetics study is of great importance in understanding the mechanism of thermal degradation reactions of biomass material. However, there have been only a few publications that investigated the kinetics of bamboo pyrolysis (Jiang et al., 2012; Mui et al., 2008). Moreover, the values of heating rates adopted in these studies were below 20 °C/min. Since heating rate has a significant effect on biomass pyrolysis, further studies on kinetics of bamboo pyrolysis are essential at higher heating rates. Kinetics analyses of microwave-induced pyrolysis of Douglas fir sawdust pellet and prairie cordgrass were performed based on response surface analysis (Ren et al., 2012; Zhou et al., 2013). Three kinetic models to describe the microwave pyrolysis of Kraft lignin were also presented using a microwave thermogravimetric analyzer (MW-TGA) (Farag et al., 2014). Nevertheless, studies on detailed biomass pyrolysis process using MW-TGA is quite few in the literature.

In this work, the bamboo pyrolysis kinetics of moso bamboo subjected to microwave and conventional heating were studied. The influence of heating rate on pyrolysis behavior of moso bamboo and the detailed comparison between microwave and conventional pyrolysis characteristics are presented. Kinetics parameters of bamboo pyrolysis based on the dominant reaction region, including the activation energy, reaction order and frequency factor, were also determined. These data can be useful for both the design and optimization of reactors and the further utilization of bamboo resources.

2. Experimental section

2.1. Materials

The bamboo sawdust (moso bamboo), obtained from a bamboo processing plant in Huzhou, China, was used as the raw material in this experiment. It had very fine particles and particles with sizes of 0.38–0.83 mm were obtained after being sieved. The proximate analysis of moso bamboo was carried out based on GB212-91 standard and it resulted in moisture, volatile matter, fixed carbon and ash contents of about 6.9, 71.3, 18.7 and 3.1 wt.% respectively. According to Omar et al. (2011), the biomass contained 23.59 wt.% hemicellulose, 44.12 wt.% cellulose and 24.49 wt.% lignin.

2.2. Experimental apparatus and procedures

2.2.1. Conventional pyrolysis

The conventional thermogravimetric tests were carried out under N_2 atmosphere with a flow rate of 50 ml/min in a Perkin Elmer Pyris-1 TGA. About 10 mg of bamboo sawdust was used in each experiment. The sample was heated from room temperature to 900 °C at heating rates of 60 °C/min, 80 °C/min, 100 °C/min, 135 °C/min and 160 °C/min and then held for about 5 min after reaching the desired temperature.

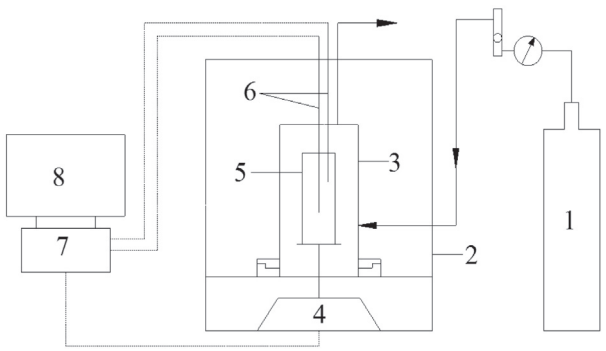
2.2.2. Microwave pyrolysis

The microwave thermogravimetric experiment was conducted using a MW-TGA. The schematic diagram of the reactor system is shown in Fig. 1. The sample was heated by a microwave oven which can be operated with the maximum power of 3600 W at a frequency of 2.45 GHz. An electronic balance (Capacity: 1 kg, Precision: 0.01 g) located below the quartz reactor was used to measure the variation of the sample mass during the test. An open quartz reactor (150 mm length, 45 mm i.d.) was used in this study. Temperature is a key parameter used in kinetic analysis and to calculate activation energy. However, accurate temperature measurement is very difficult under microwave field because of the non-uniform distribution of electric field intensity. In this study, two stainless steel-sheathed type K thermocouples (0–800 °C) with a diameter of 3 mm were used and the thermocouples sheath were earthed to avoid arcing. One thermocouple was inserted to a point near the center of bamboo bed to monitor the temperature of inner part of the bed and the other was at a point of the upper layer of the bed. The temperature of the upper layer (T_o) and the temperature of center of the bed (T_i) were the average value of three repeated experiments. The average reaction temperature used for the kinetic study was the average value of T_o and T_i.

About 50 g samples were placed in the open quartz reactor, which in turn was placed on a PTFE tray inside a quartz container. In order to maintain an inert atmosphere, the oven was purged with nitrogen gas at a flow rate of 4 L/min prior to the microwave treatment as well as during the experiment. Microwave power was selected at 800 W and the heating time was 10 min. The temperature and weight data are automatically collected and recorded by the data acquisition system during the experiments. All experiments were carried out in three runs to confirm the values obtained.

2.3. Pyrolysis kinetics

The pyrolysis process of biomass can be expressed as the following reaction scheme:



(1) Nitrogen, (2) Microwave oven, (3) Quartz container, (4) Balance, (5) Quartz reactor, (6) Thermocouple, (7) Data acquisition system, (8) Computer

Fig. 1. Schematic diagram of experimental set-up.

Bamboo → volatiles + solid residue

The decomposition rate of moso bamboo is generally described as follow:

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \quad (1)$$

where T is the absolute temperature (K); $k(T)$ is the rate constant dependent on temperature; α is the fractional conversion at time t(s); $f(\alpha)$ is a function depending on the reaction mechanism. In this study, the fractional conversion was defined as:

$$\alpha = \frac{(w_0 - w)}{(w_0 - w_\infty)} \quad (2)$$

where w_0, w and w_∞ are the initial, time t and final weight of the sample, respectively. The rate constant is normally described by the Arrhenius equation:

$$k(T) = A\exp\left(-\frac{E}{RT}\right) \quad (3)$$

where A is the pre-exponential factor (s^{-1}), E is the apparent activation energy (J mol^{-1}), and R is the gas constant (8.314 J $mol^{-1}K^{-1}$). $f(\alpha)$ is usually expressed as follow:

$$f(\alpha) = (1-\alpha)^n \quad (4)$$

where n is the reaction order. The heating rate is defined as:

$$\beta = \frac{dT}{dt} \quad (5)$$

Combination of Eq. (1) and Eqs. (3–5) gives:

$$\frac{d\alpha}{dT} = \frac{A}{\beta}\exp\left(-\frac{E}{RT}\right)(1-\alpha)^n \quad (6)$$

Eq. (6) is the elementary expression for calculating kinetic parameters based on the TGA data.

In this study, Coats–Redfern method (Coats and Redfern, 1964) was applied to determine the kinetic parameters. Re-arranging and integrating Eq. (6) gives:

$$\int_0^\alpha \frac{d\alpha}{(1-\alpha)^n} = \int_{T_0}^{T}\frac{A}{\beta}\exp\left(-\frac{E}{RT}\right) \quad (7)$$

Integrating both sides followed by taking the logarithm of the obtained equation leads to:

$$\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right] = \ln\left[\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right] - \frac{E}{RT} \quad (\text{for } n=1) \quad (8)$$

and,

$$\ln\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right] = \ln\left[\frac{AR}{\beta E}\left(1-\frac{2RT}{E}\right)\right] - \frac{E}{RT} \quad (\text{for } n\neq 1) \quad (9)$$

Due to $2RT/E \ll 1$, simplifying Eqs. (8) and (9) gives:

$$\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right] = \ln\left[\frac{AR}{\beta E}\right] - \frac{E}{RT} \quad (\text{for } n=1) \quad (10)$$

and,

$$\ln\left[\frac{1-(1-\alpha)^{1-n}}{T^2(1-n)}\right] = \ln\left[\frac{AR}{\beta E}\right] - \frac{E}{RT} \quad (\text{for } n\neq 1) \quad (11)$$

Thus, a plot of $\ln[-\ln(1-\alpha)/T^2]$ against $1/T$ when $n = 1$ or $\ln\left[(1-(1-\alpha)^{(1-n)})/(T^2(1-n))\right]$ versus $1/T$ when $n \neq 1$ should be a straight line with slope ($-E/R$) and intercept ($\ln[AR/\beta E]$) for the correct value of n. The kinetic parameters were determined by combining iterative and the least squares method.

3. Results and discussion

3.1. Conventional pyrolysis of moso bamboo using TGA

Firstly, conventional thermogravimetric experiments were carried out at heating rates of 60, 80, 100, 135 °C/min. It was found that the pyrolysis process could be divided into four stages. In the first stage (<180 °C), a slight weight loss was observed, due to the dehydration within samples. In the second stage (180–220 °C), the macromolecules of bamboo were activated by the glass transition and depolymerization of biomass components (Bradbury et al., 1979), and there was scarcely any weight loss of sample. Different from the first two stages, samples underwent a fast thermal decomposition in the third stage (220–530 °C). Most of the hemicelluloses and cellulose and partial lignin were decomposed, resulting in 77 wt.% of the sample weight loss and the maximum reaction rate obtained at the temperatures ranging from 370 to 400 °C. In the last stage (>530 °C), the pyrolysis of the residues of bamboo was very slow. This can be ascribed to the degradation of lignin residues or tar and char from the third stage (Jiang et al., 2012).

With an increase in heating rates from 60 to 135 °C/min, the maximum reaction rates were increased but slightly shifted towards higher temperature regions.

According to the above mentioned, it could be due to the heat-transfer effect on the particle (Mui et al., 2008). Thus, a higher heating rate would result in a larger temperature difference between the interior and external of the particle and led to the thermal decomposing of the inner part of particle and a quicker release of volatiles at higher temperatures.

The kinetic parameters for the pyrolysis of moso bamboo at different heating rates were determined by Coats–Redfern method based on the primary reaction region and the results are shown in Table 1. The activation energy varied from 58.30 to 84.22 kJ/mol with a decrease in the heating rate, much lower than the value of more than 150 kJ/mol obtained by Jiang et al. (2012) for moso bamboo pyrolysis at a heating rate of 10 °C/min. This indicated that thermal decomposition of moso bamboo could be performed more easily at high heating rates because lower activation energy indicated less amount of energy required to start the chemical reaction. It was also found that the reaction order decreased from 2.80 to 1.00 with heating rates increasing from 60 to 135 °C/min, revealing a greater impact of the volatile content within the bamboo on the pyrolysis reaction rate at lower heating rates.

3.2. Microwave pyrolysis of moso bamboo

The efficiency of biomass pyrolysis under microwave irradiation is dependent mainly on its dielectric property, which is determined by its chemical compound and, to a lesser extent, by it physical structure (Fernández et al., 2011). As biomass material generally has poor dielectric properties, sufficient microwave input power is required to initiate the thermal decomposition of biomass (Robinson et al., 2010). In this work, microwave pyrolysis of moso bamboo was carried out at an input power of 800 W and a heating time of 10 min. The average temperature, residual weight rate and

Table 1
Kinetic parameters obtained from conventional pyrolysis at different heating rates.

β (°C/min)	T (°C)	E (kJ/mol)	$\ln A$ (s^{-1})	R^2	n
60	284.5–433.9	84.22	16.38	0.988	2.80
80	299.0–456.6	75.10	14.53	0.995	2.20
100	304.0–466.7	73.85	14.18	0.998	1.70
135	270.0–475.0	58.3	11.28	0.999	1

DTG of moso bamboo as a function of time are presented in Fig 2. It can be seen that the average temperature of sample rose rapidly to about 125 °C in about 1.3 min mainly due to the heating of moisture. After that, the temperature experienced a slow increase from about 125 to 135 °C because the tangent loss factor of sample (which indicates the ability of materials to heat under microwave fields) decreased due to the moisture evaporation (Motasemi et al., 2014). Then, the temperature almost exhibited a liner increase from 135 to 545 °C in about 2.7 min and subsequently remained basically stable at about 590 °C because of the balance between heat loss and heat provided by microwaves. Accordingly, the pyrolysis of moso bamboo underwent a moisture evaporation process below about 135 °C, and a quick thermal decomposition process was observed in the temperature range of 175–500 °C with an average heating rate of about 160 °C/min, followed by a slow weight loss process until the end of the heating.

In order to make a comparison, the pyrolysis of moso bamboo was also conducted at a heating rate of 160 °C/min using conventional TGA. The variations of temperature, the residual weight rate and DTG of moso bamboo as a function of time were presented in Fig 3. Compared to the results in Fig 2, a similar TG curve was observed except but the obvious glass transition stage shown in conventional pyrolysis was absent here. In other words, the decomposition of moso bamboo took place instantly after the completion of moisture evaporation, or the first two stages shown in microwave pyrolysis were partially overlapped. Meanwhile, the rapid pyrolysis started at a lower temperature than that for conventional pyrolysis. The differences created were probably by the microwave thermal effects. Hotspot is a thermal effect in heterogeneous reactions that can be generated by the non-uniform distribution of electromagnetic field or selective heating of microwaves for the solid-to-vapor conversion (De la Hoz et al., 2005). It can lead to local regions within the sample having a higher temperature than the average temperature. For biomass material, the selective heating could probably take place because of the presence of metal ions. The hotspot regions are not representative of the reaction conditions as a whole. Hence, the presence of hotspots can make some reaction to occur seemingly at a lower temperature.

It is worthwhile to note that the weight loss rates were faster under microwave irradiation than those observed under conventional pyrolysis when reaction temperature was lower than 360 °C. The result indicated that pyrolysis reaction could be accelerated under microwave heating at low temperatures. This might be due to the volumetric heating style of microwaves, which resulted in reductions in temperature gradients within sample and heat loss: more heat in the sample facilitates chemical

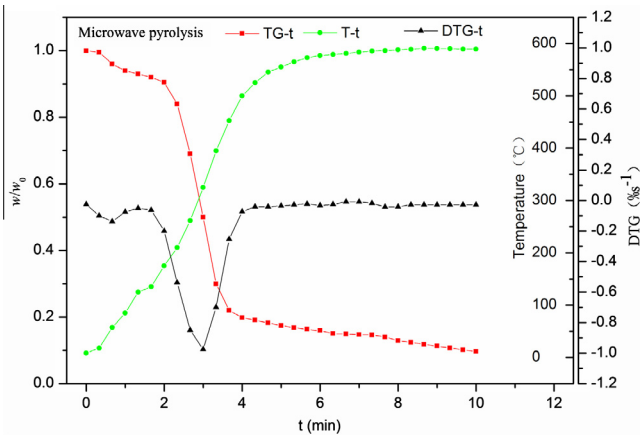


Fig. 2. TG–*t*, *T*–*t*, and DTG–*t* curves during microwave pyrolysis process.

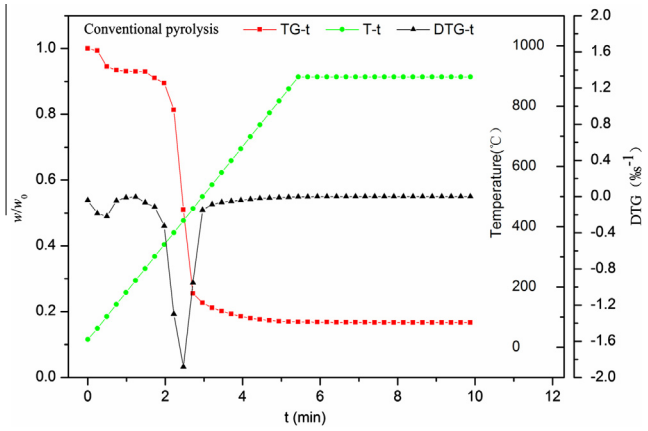


Fig. 3. TG–*t*, *T*–*t* and DTG–*t* curves during conventional pyrolysis process with a heating rate of 160 °C/min.

reactions (Sun et al., 2012). However, the maximum weight loss rate was lower during microwave pyrolysis than that under conventional heating. It is probably because that the stacking of mass samples retarded the escape of volatiles during microwaves pyrolysis process. The postponed escape inevitably affected the partial pressure nearby particles, and ultimately had an effect on the pyrolysis process.

As shown in Section 2.3, the values of activation energy and frequency factor were calculated based on a constant heating rate during the pyrolysis process. The temperature showed an approximately linear increase with a heating rate of about 160 °C/min in the range of 175–500 °C for microwave pyrolysis. Thus, the kinetic parameters were determined using the data of this region. For the conventional pyrolysis using TGA, the main pyrolysis reaction occurred in the temperature range of 300–500 °C. Table 2 presents the calculated kinetic parameters for both heating methods. For microwave pyrolysis, the differences in activation energy for each run were all less than ±9% of the value obtained from the average of all the measured temperatures. The value of activation energy should be the same under the same heating rates if the kinetic processes in both cases are the same (Sun et al., 2012). In fact, the activation energy was dramatically lower under microwave irradiation than that under conventional heating, revealing that kinetic processes for both heating methods were different. It was found that the value of frequency factor was also significantly affected by microwaves, and decreased dramatically compared to that for conventional pyrolysis. The decrease in frequency factor means a negative effect of microwaves on the reaction rate. However, the lowered activation energy could compensate this negative effect well.

3.3. Kinetic compensation effect

In this experiment, a linear dependence between the values of $\ln A$ and E ($\ln A = 0.18882E + 0.24895$; $R^2 = 0.988$) has been observed for both microwave and conventional pyrolysis of moso bamboo. The high linearity between $\ln A$ and E suggests the existence of kinetic compensation effect. No consensus on

Table 2
Kinetic parameters obtained from both conventional and microwave pyrolysis processes.

Pyrolysis method	T (°C)	E (kJ/mol)	$\ln A$ (s^{-1})	R^2	n
Conventional pyrolysis	300–500	49.6	8.87	0.953	0.9
Microwave pyrolysis	175–500	24.5	5.286	0.972	1.5

the occurrence of kinetic compensation effect has yet been reached, although many have been proposed (Galwey, 2004). However, it is possible that the introduction of compensation effect may act as a tool for check-up in the kinetic calculation for materials of similar reactivity (Mui et al., 2010). Here, the kinetic parameters for microwave pyrolysis have been successfully incorporated into the compensation effect. The result showed that the calculated kinetic parameters for microwave pyrolysis were rational to a certain extent.

4. Conclusions

In this work, the kinetics of moso bamboo pyrolysis was investigated using a conventional TGA and a MW-TGA. The results indicated that heating rate had a significant effect on the biomass pyrolysis process. Compared with conventional heating, the activation energy is much lower for microwave pyrolysis. This might be due to the volumetric heating style of microwaves. A kinetic compensation effect between $\ln A$ and E was observed. The high disposal efficiency and low activation energy suggested that microwave heating would be a more promising technology than conventional heating for biomass pyrolysis.

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